

Title: Tagged Composites Mechanical Properties Investigation and Correlation with Fractographic Examination

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ABSTRACT1

The mechanical properties of smart composites with embedded ferromagnetic tagging were examined. Flexure and tension tests were performed per ASTM standards. Weakening of the fiber/matrix interface resulting in a reduced interfacial shear strength, and reduced mechanical properties due to magnetite tagging powders was initially observed. Macroscopic and microscopic observations (fiber pull-out length, matrix adhesion remains on the fiber surface, etc.) were corroborated with the observed mechanical properties. An improved tagging system, using dried lignosite powder, gave much better mechanical properties, and indicated that adequate materials and process control can produce good tagged GFRP composites.

EXPERIMENTAL PROCEDURE

Smart composite samples "tagged" with magnetic substances (magnetite and lignosite solution) were tested. Pultruded C-channel profiles used as ladder rails (1"×3.5", color code yellow) were manufactured by Creative Pultrusions, Inc., and have a five layer internal architecture. The outside and center layers were thin random weaved mats. Thicker unidirectional pultruded E-glass fiber layers were contained between the mats in a vinyl ester resin. The tagging consisted of 2% ferrous oxide (magnetite) particles dispersed predominately in the outside two layers. Testing was done in a Instron 4505 machine with 1 kN and 20,000 lb capacity load cells run under displacement control through the GPIB interface using a Macintosh Power PC and LabVIEW software. The mechanical data was downloaded, stored and analyzed in the same computer. ASTM type specimens were prepared from the C-channels and instrumented with CEA-06-125UW-350 strain gages.

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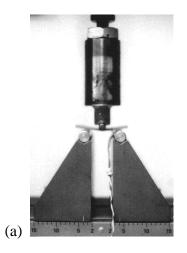




Figure 1 Test fixtures: (a) flexure test fixture; (b) tension test fixture

Three-point bending tests were done per ASTM D790 (Figure 1a). Specimen dimensions were $60\times25\times2.4$ mm³. Ten specimens of each type were used. Each specimen was straingauged in the center, and then centered on the 3-point bend support, with the strain gage on the under side. The cross-head applied the load at 1 mm/min until crack initiation.

Tension test were done per ASTM D3039 (Figure 1b). Six tension specimens of each type were cut to $100 \times 13 \times 2.4 \text{ mm}^3$ dimensions from the same structural C-channel members and strain gauged on both side at the center position. The specimens were placed into vise grips (using 60 grit sandpaper to increase friction) and loaded to failure at 1.5 mm/min load rate.

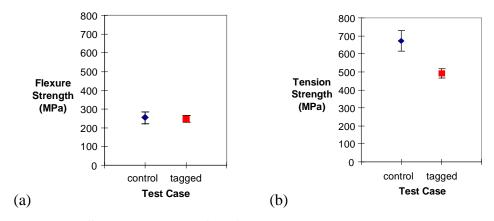


Figure 2 Strength results for C-channel tagged and control composites: (a) flexural strength; (b)tensile strength

MECHANICAL TESTING RESULTS

The load, displacement, and strain data was processed to yield the strength data (Figure 2). Figure 2a shows a 3% decrease in flexural strength of the tagged composite, which can be considered within the experimental error. This conclusion is similar to that of Linares (1996), and of Quattrone, Berman, and Voyles (1996). Figure 2b shows a significant decrease in tension strength (26.9%) and in the elongation (25%).

Table 1. Comparative results of the mechanical strength data.

		Linares (1996). Reichhold Chemicals	Quattrone, Berman, and Voyles (1996). CERL	Giurgiutiu and Jerone (1996) Virginia Tech
Flexure Strength ¹	Control	82.1	67.6	36.3
(ksi)	Tagged	78.2 (-4.6%)	65.7 (-2.7%)	35.3 (-2.8%)
Tension Strength	Control	112.0	105.7	96.0
(ksi)	Tagged	72.6 (-35.2%)	72.1 (-31.7%)	70.1 (-26.9%)

¹Flexure strength variation from investigator to investigator is attributed to specimen length effects.

This decrease is consistent with the finding of Linares (1996) who reported a 35.2% decrease in strength and a 36% decrease in elongation, and of Quattrone, Berman, and Voyles (1996) (31.7% decrease in both quantities). A summary of all these results is presented in Table 1. The conclusion of Table 1 is that tagging with magnetite powders significantly reduces the tension strength but not the flexure strength. The explanation of these difference lies in the effect of interlaminar shear strength and of internal fiber architecture of the C-channel profile, where the high-strength glass fiber tows are placed in the middle of the thickness, while the outer layers are made of low-strength glass mat and veil material. Figure 3 shows the post-failure aspect of the untagged (control) and tagged tension specimens. It is apparent (Figure 3a) that the aspect of the untagged (control) specimen corresponds to wide spread catastrophic failure at high energy levels. Figure 3b shows the tagged specimen, which seems to show a "clean" cut in the outer veil and slippage between the thin outer mats (which has higher tagging concentration) and the inner unidirectional fiber layers. In fact, the load transfer path between the loading grips and the inner high-strength core of the specimen has been interrupted by the loss of adhesion in the interface.

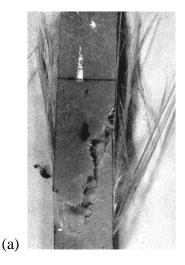




Figure 3 Comparative macroscopic aspects of tension failure: (a) control specimen; (b) tagged specimen

MICROSCOPIC OBSERVATIONS

Fiber matrix adhesion can be correlated to the pull-out length of a broken fiber (Figure 4a). Following Giurgiutiu *et al.* (1996), the equations controlling the fiber pull-out process are:

$$P(x) = 2\pi \cdot r_f \cdot x \cdot \tau_0 \quad , \qquad P_{\text{max}} = \pi \cdot r_f^2 \cdot X_f \quad , \qquad \delta = \frac{r_f \cdot X_f}{2 \cdot \tau_0} \tag{1}$$

The pull-out fiber breaks when the built-up load P(x) reaches the ultimate fiber load, P_{max} . The pull-out fiber length varies inversely to the fiber-matrix bonding strength. Since the control and tagged composite were constructed from the same materials, the predominate variable was the interfacial shear stress τ_0 . Equation (1) indicates that a longer pull-out length, i. e., a greater δ , indicates a lower τ_0 , and a weaker fiber-matrix interface. Therefore, according to this fiber-pull-out argument, the fibers resulting from the composite with a lower strength (i. e. the tagged composite specimens), should be longer and "cleaner" than the fibers from the control composite specimens.

To verify the correlation with the fiber-pull-out-length, the specimens were analyzed at the microscopic level. The microscopic work was done with a ISI SX-40 Scanning Electron Microscope (SEM). Each specimen was gold coated using a sputtering machine. Figures 4b and 4c are representative micrographs for cracked surface edge in both specimens. The fibers are 25% longer in the tagged composite, which, according to Equation (1) infers a 25% decrease in interfacial shear strength.

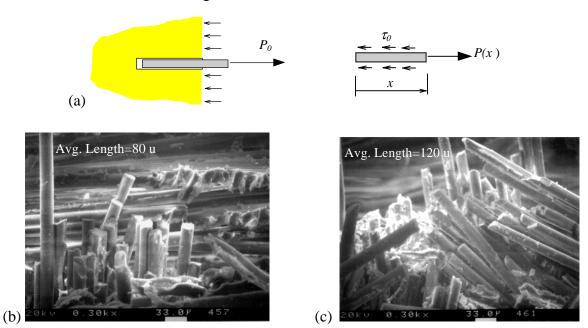
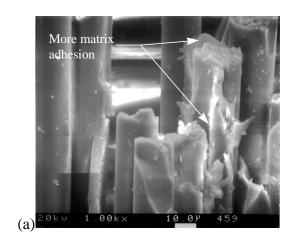


Figure 4 Microscopic examination of pull-out fibers at ×300 magnification:
(a) Schematic drawing of pull-out fiber force and interfacial shear stress;
(b) control composite specimens; (c) tagged composite specimens.



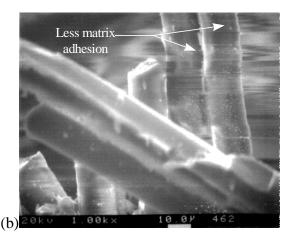


Figure 5 Microscopic examination of pull-out fibers at x1000 magnification: (a) control composite specimens; (b) tagged composite specimens.

Since the bonding of the resin to the fibers is weaker in the tagged composite than in the control, it is expected that the tagged fibers should have less matrix adhered to them. This difference is evident at higher microscopic magnification (×1000). Figure 5 compares the surface morphology of the untagged (control) composite (Figure 5a) with that of the tagged composite (Figure 5b) which presents significantly lower adhesion on the fibers surface.

IMPROVED MECHANICAL PROPERTIES THROUGH LIGNOSITE TAGGING

A tagged composite with greatly improved mechanical properties was recently presented and tested (Pauer, Gauchel, Klett and Troutman, 1996). This improved tagged composite material used lignosite powder (4% by weight of resin) during the pultrusion process and produced C-channel profile ladder rails dimesionally similar to those previously tagged with magnetite particles, but colored with blue pigment.

Mechanical testing of these improved tagged composites, were performed at the US Army CERL facilities. The results, shown in Table 2, indicate the advantages of the improved tagging formulation. As with the previous tagged material, the differences between the "tagged" and "untagged" flexure strengths is within the experimental error. As compared with the previous tagged material, the difference in the tensile strength is much smaller (10%), thus indicating the advantages of the lignosite tagging formulation.

Table 2. Comparative results of the mechanical strength data for the improved tagged composite formulation.

	Untagged (control)	Tagged
Flexure strength	68.5±0.5 ksi	66.3±2.3 ksi
Tensile strength	61.0±1.0 ksi	54.7.0±1.0 ksi

CONCLUSIONS

The most significant finding of these tests lies in establishing the micro-mechanical mechanism that lead to the substantial (25-35%) decrease in the tension strength observed in the tagged composite samples using the magnetite tagging formulation. This difference was explained in view of the internal fiber architecture and fiber/matrix and interlaminar bonding strength. The load transfer path under tension loading, from machine grips, through the outside mat and veil layers to the inner high-strength unidirectional fiber layers was identified to be primarily adhesive. With a weakened adhesion strength, the load could be not properly transmitted to the inner high-strength layer, and the overall tension strength of the composite decreases. This difference in bonding strength could be correlated with the different aspect of the failed specimens, as illustrated in Figure 3. Further examination at microscopic level revealed further correlation. The tagged composite fibers had less resin still attached to the broken fibers, and the pull-out length of these broken fibers were, on average, 25% longer than the control fibers. This finding suggests a 25% decrease in the interfacial shear stress, which corresponds to a 25% decrease in strength, similar to the mechanical testing results.

The fact that the flexure strength did not present a similar significant decrease was also explained. In the flexure test, the loading was more directly distributed in the high-strength inner layers, since the thin and weak outside layers are not expected to carry much load. Hence, the further weakening of the outside layers due to tagging did not significantly affect the overall flexure strength of the composite. Additionally, as determined by Quattrone, Berman and Voyles, (1996) the tagging was not uniform across the thickness, and hence only affects the outer, already weak, layers. Further weakening of these layers by the application of tagging does not significantly modify the resulting flexure strength.

The latest results obtained with an improved tagging formulation utilizing lignosite material have shown that most of the initial problems have been alleviated. The decrease of the tensile strength has been reduced to 10%, thus indicating that adequate materials and process control can produce good tagged GFRP composites.

Acknowledgments

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